1974

# NDE Prediction of Adhesive Bond Failure Areas

Tennyson Smith
*Rockwell International*

# NDE Prediction of Adhesive Bond Failure Areas

**Abstract**
Suppose you want to find out if an adhesive joint is going to fail before you ever bond it together. Then you can do something about it before the bond is made and that is the kind of information that we are working on here. Consider an assembly line in a factory where you have surface preparations going on, and finally, you get to the point where an adhesive bond is going to be made. It would be very useful if just prior to bonding you had a technique for monitoring the surface to tell you whether the surface is, indeed, a proper surface for the bonding procedure. Any number of things could go wrong upstream; for example, you might have run out of solution in a tank or a tank had become contaminated.

**Disciplines**
Materials Science and Engineering | Structures and Materials

# NDE PREDICTION OF ADHESIVE BOND FAILURE AREAS

Tennyson Smith
Science Center, Rockwell International
Thousand Oaks, California

Suppose you want to find out if an adhesive joint is going to fail before you ever bond it together. Then you can do something about it before the bond is made and that is the kind of information that we are working on here. Consider an assembly line in a factory where you have surface preparations going on, and finally, you get to the point where an adhesive bond is going to be made. It would be very useful if just prior to bonding you had a technique for monitoring the surface to tell you whether the surface is, indeed, a proper surface for the bonding procedure. Any number of things could go wrong upstream; for example, you might have run out of solution in a tank or a tank had become contaminated.

We are looking at surfaces with three different techniques; ellipsometry, surface potential difference and photo-emission (see Fig. 1). We map surfaces prior to the adhesive bond procedure, and use the map to tell us where the contamination is. After bonding the samples together, and fracturing them, we look at them again, and find out if failure actually occurs where the contamination was. By that procedure we are able to demonstrate that we, indeed, can detect in advance where the failure will occur.

I would like to show you first just a very short summary of the three techniques I mentioned.

One uses an ellipsometer which is very sensitive to films on the surface. Another is photo-emission detection in air where we just shine UV light onto the sample and collect the electrons, and the sample is moved back and forth past the detector to map the surface. We also make

surface potential difference measurements which measure the difference in potential between the reference electrode and the surface. We thus have three independent measurements that are very sensitive to surface properties. If anyone wants to know more details, I will be glad to tell them later. I don't have time at this point.

We have a number of samples that were deliberately contaminated in certain areas in the following ways as given in Table I. The resulting bond strengths are also given in the table. The samples are aluminum 2024-T3 and an epoxy adhesive in a double over-lap shear joint. They had been given the FPL etch and adhesively bonded. The control gives about 3,850 psi, and when we put a fingerprint on one of them, just a dry fingerprint, we see we actually got better bonds. If we put one corner of the sample in water, some contamination from the water surface was transferred and the bond strength dropped down 300 psi. If we deliberately put on two monolayers of erucic acid to stimulate some organic matter, it dropped down about 300 psi, and if we put on three monolayers, we got about the same value. If we put a fingerprint on where the finger had been dipped in some silicone grease and then wiped off, the bond strength dropped down to 2,600 psi. All I am showing here is that by deliberately contaminating the surface, we do see the bond is degraded in most cases.

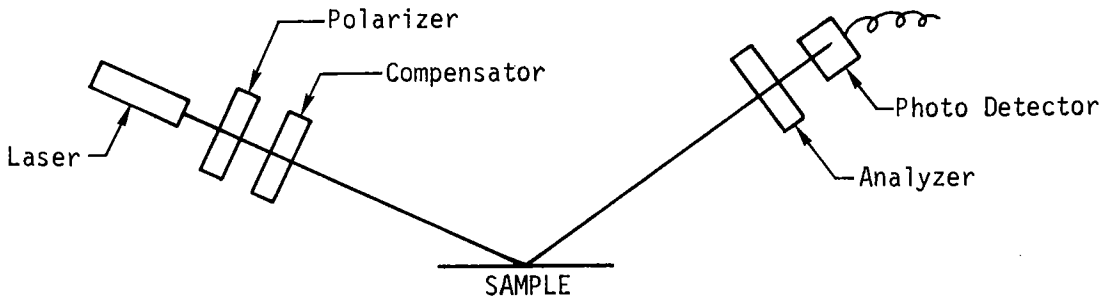
Now, the next thing we wanted to do was see if we could find the contamination areas prior to bonding. Figure 2a is an example where we show the map obtained by photo emission as the sample was scanned. The current is zero with the collector on either side of the sample and approaches steady state negative value over the control sample. You can see the map is pretty well uniform across the control sample with no contamination.

Now, if you dip one corner of the sample into water (Fig. 2c), and then map it with the photo-emission, you see the map reveals the small amount of contamination that comes from the water surface (Fig. 2b). If you put one corner into water that has erucic acid on the surface so you deposit a couple of monolayers of erucic acid, the photo-emission map reveals the triangle shaped area very well in Fig. 2d.

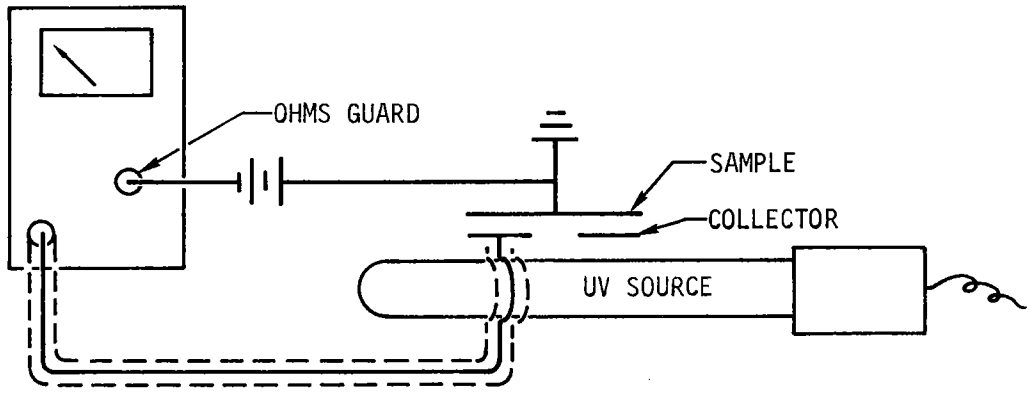
TABLE I

Bond Strength of a 2024-T3 - Epon 934 Couple
After Deliberate Contamination

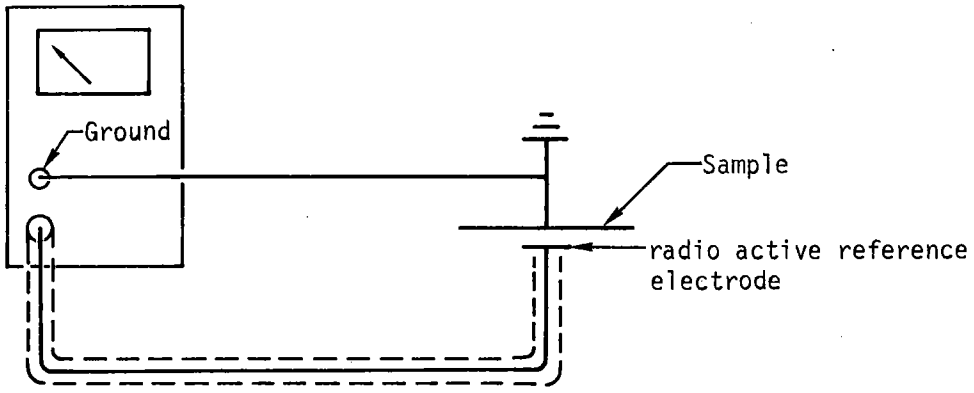
| Contamination | Bond Strength |
|---|---|
| | PSI |
| Control | 3850 |
| Finger Print | 3960 |
| Contaminated Water | 3570 |
| 2 Monolayer Erucic Acid | 3520 |
| 3 Monolayer Erucic Acid | 3530 |
| Finger Print Silicone Grease | 2600 |




(a) ELLIPSOMETRY




(b) PHOTO-EMISSION




(c) SPD

Fig. 1. Techniques for surface characterization.

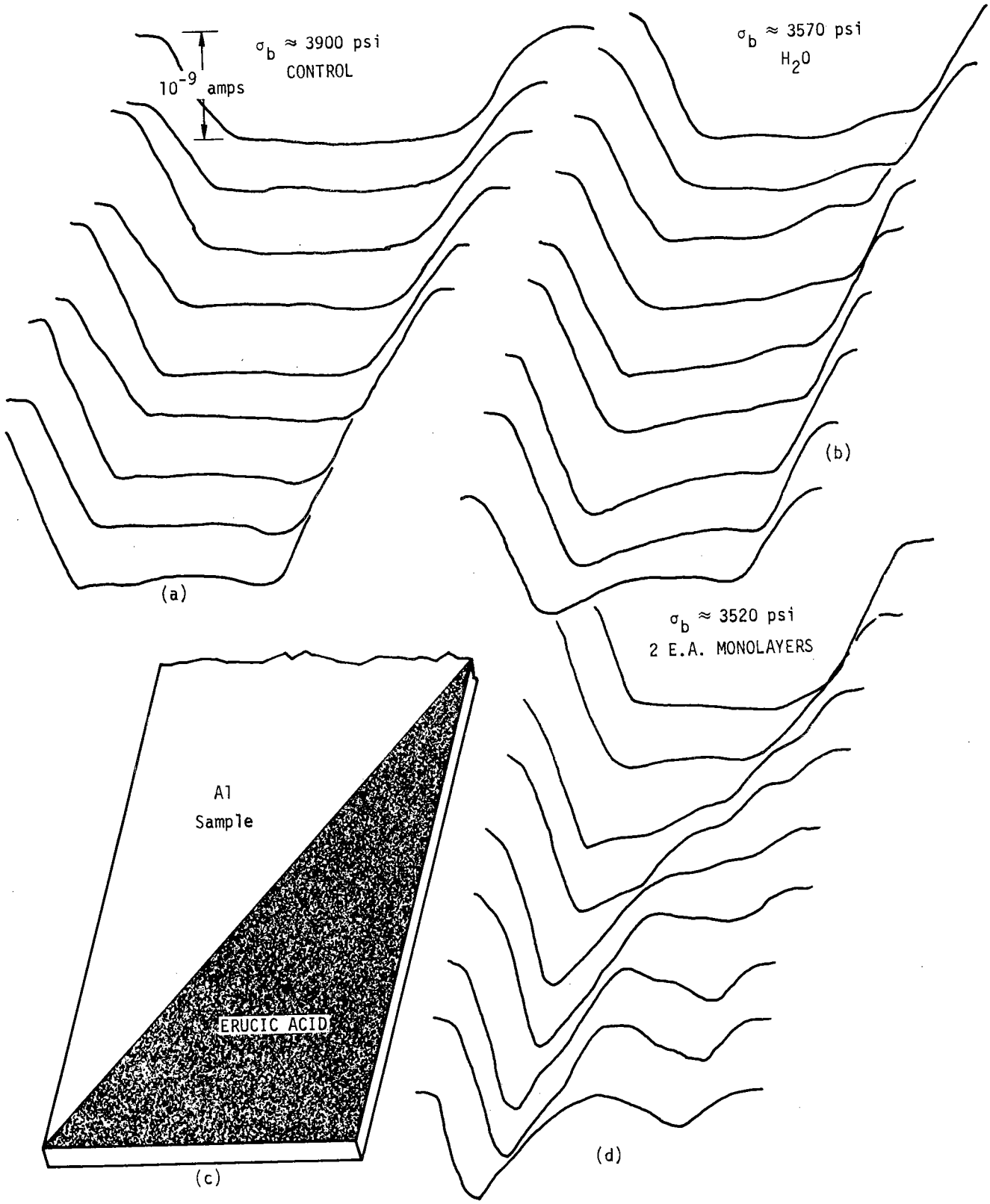



Fig. 2. Photo-emission maps.

The same kind of map with respect to surface potential is shown in Fig. 3. The higher resolution in Fig. 3b and d as compared to Fig. 3a, was obtained with a smaller reference electrode. The controlled experiment yields a pretty uniform map for surface potential measurements. When you dip the sample in the water, you still have a fairly uniform map and you don't really see the contamination like you did with the photo emission, but if you put on erucic acid then you can see that the surface potential extremely well delineates the surface contamination in Fig. 3d.

Now, in the case of the fingerprint, Fig.4 is a map of the photo emission for the control (Fig. 4a) and the dry fingerprint (Fig. 4b). The surface potential map (Fig. 5) also shows the fingerprint. You can see the shape of the fingerprint on the ellipsometric map - Fig. 6. Figure 6 only shows the reflectance at one azimuth setting of the analyzer. Similar plots at two other azimuths allow calculation of a film thickness map.

In conclusion, then, it appears that if you had one of these instruments scanning parts that were coming down the assembly line and you could detect parts or areas of parts that were contaminated, then process control for bond strength would be greatly enhanced. If the signal from one of the instruments exceeded the bounds for a proper surface preparation, a red light comes on or some warning is given so you know that something has gone wrong, and you can go back and adjust the appropriate process parameter.

As is apparent, this work is in its early stages. What has to be done next is to demonstrate for other kinds of problems that will arise in the field that such maps exist and what the accept/reject criteria are. That will take some more research, but I think we have demonstrated that it certainly looks like a very feasible thing to do.

Thank you.

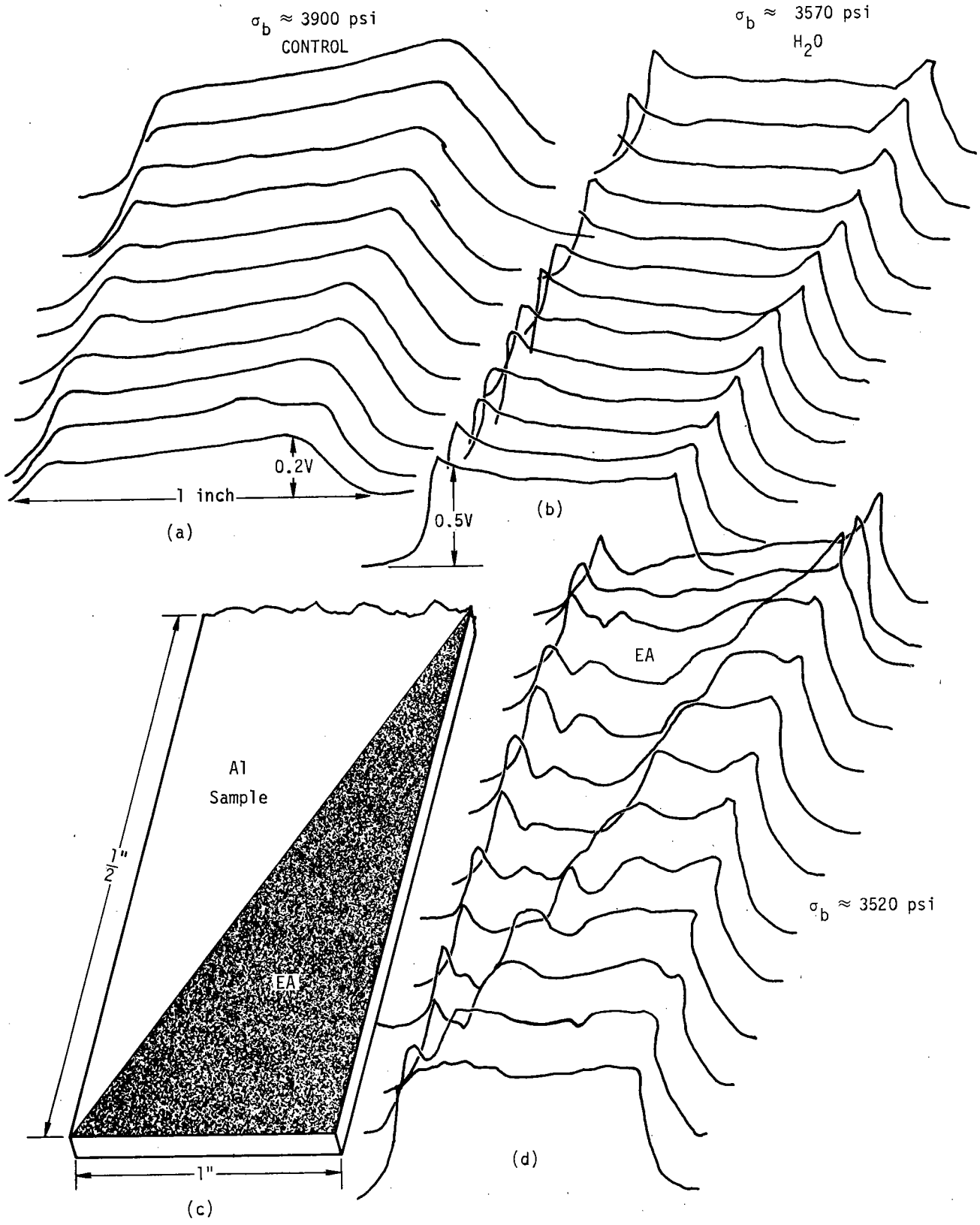



Fig. 3. Surface Potential Difference maps.

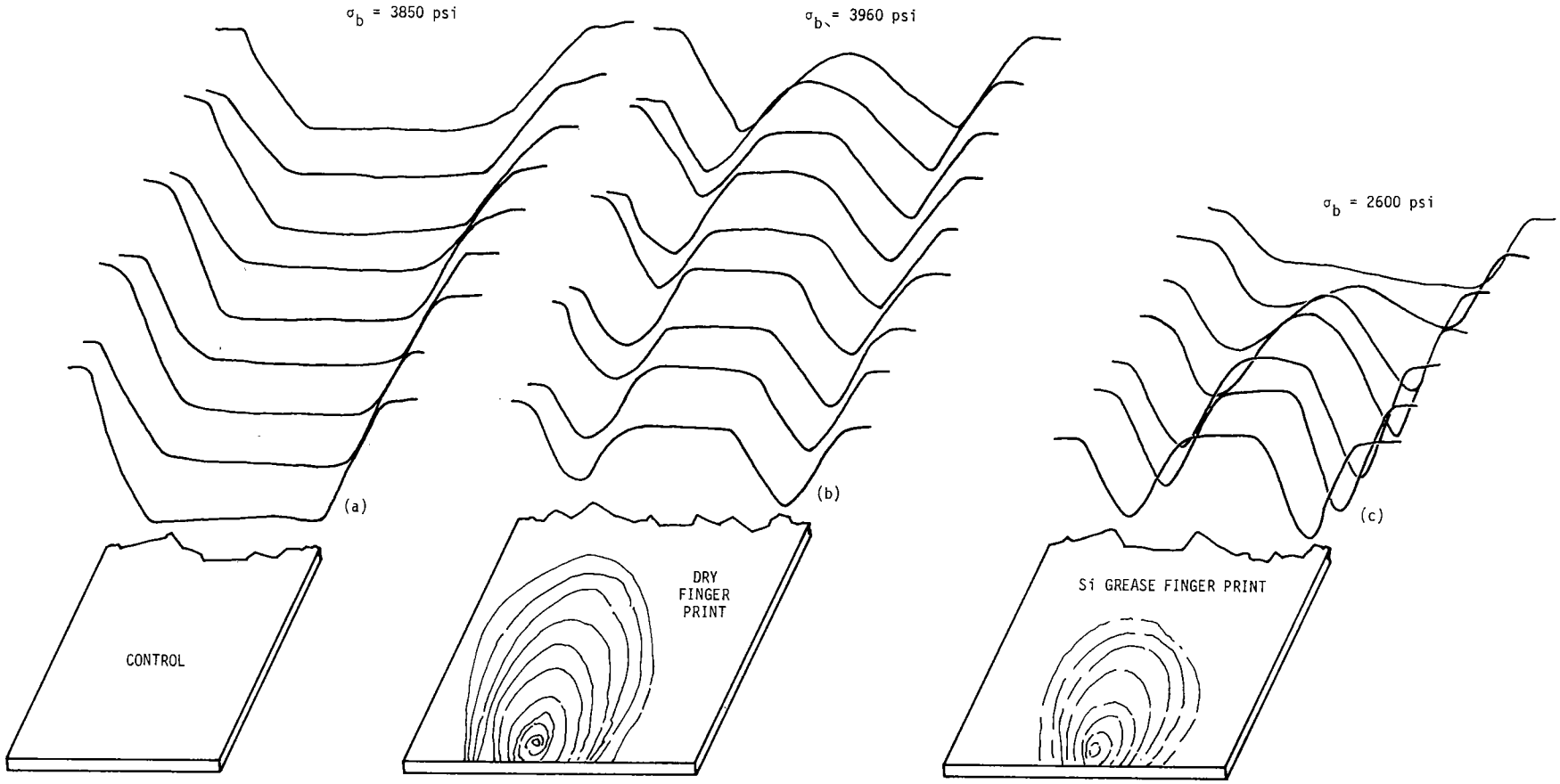



Fig. 4. Photo-emission maps of the control, a dry finger print and a greasy finger print.

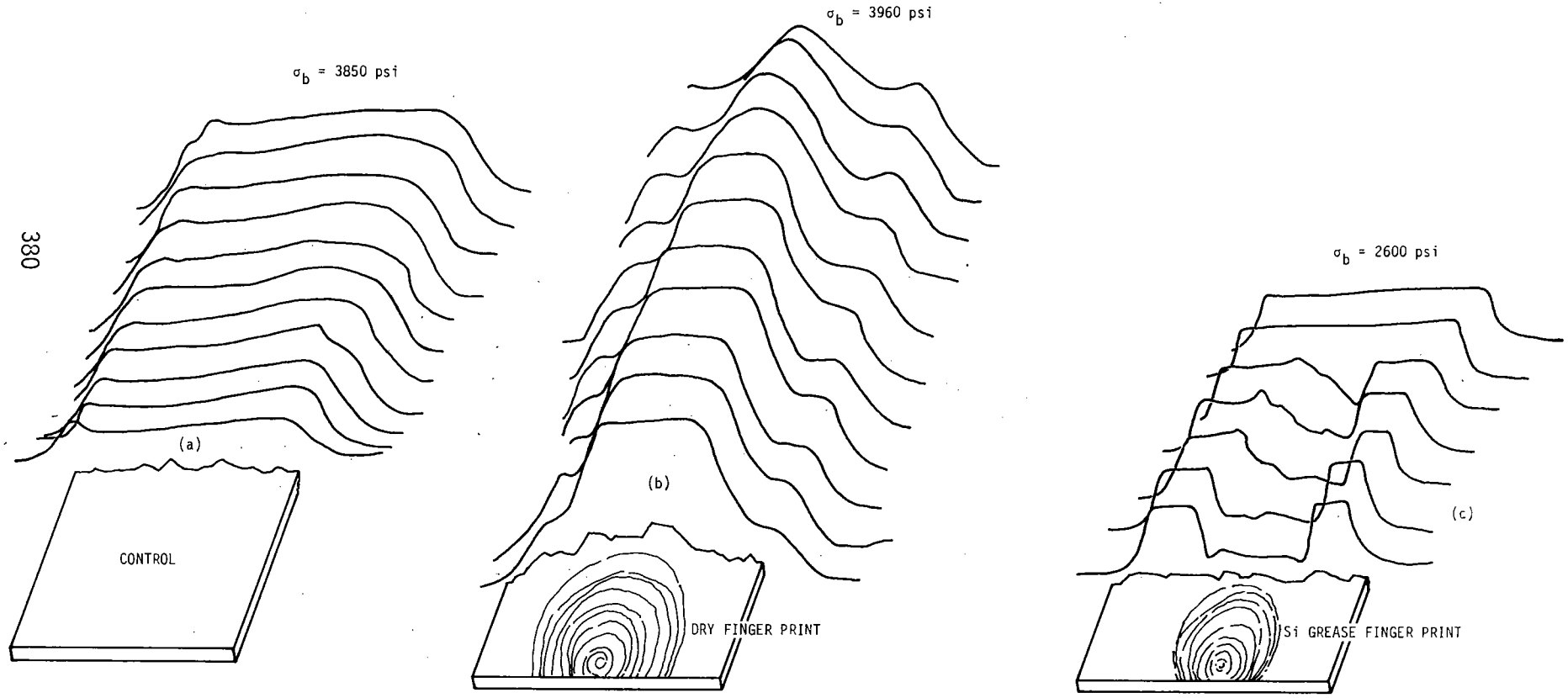



Fig. 5. Surface Potential Difference maps of the control, a dry finger print and a greasy finger print.

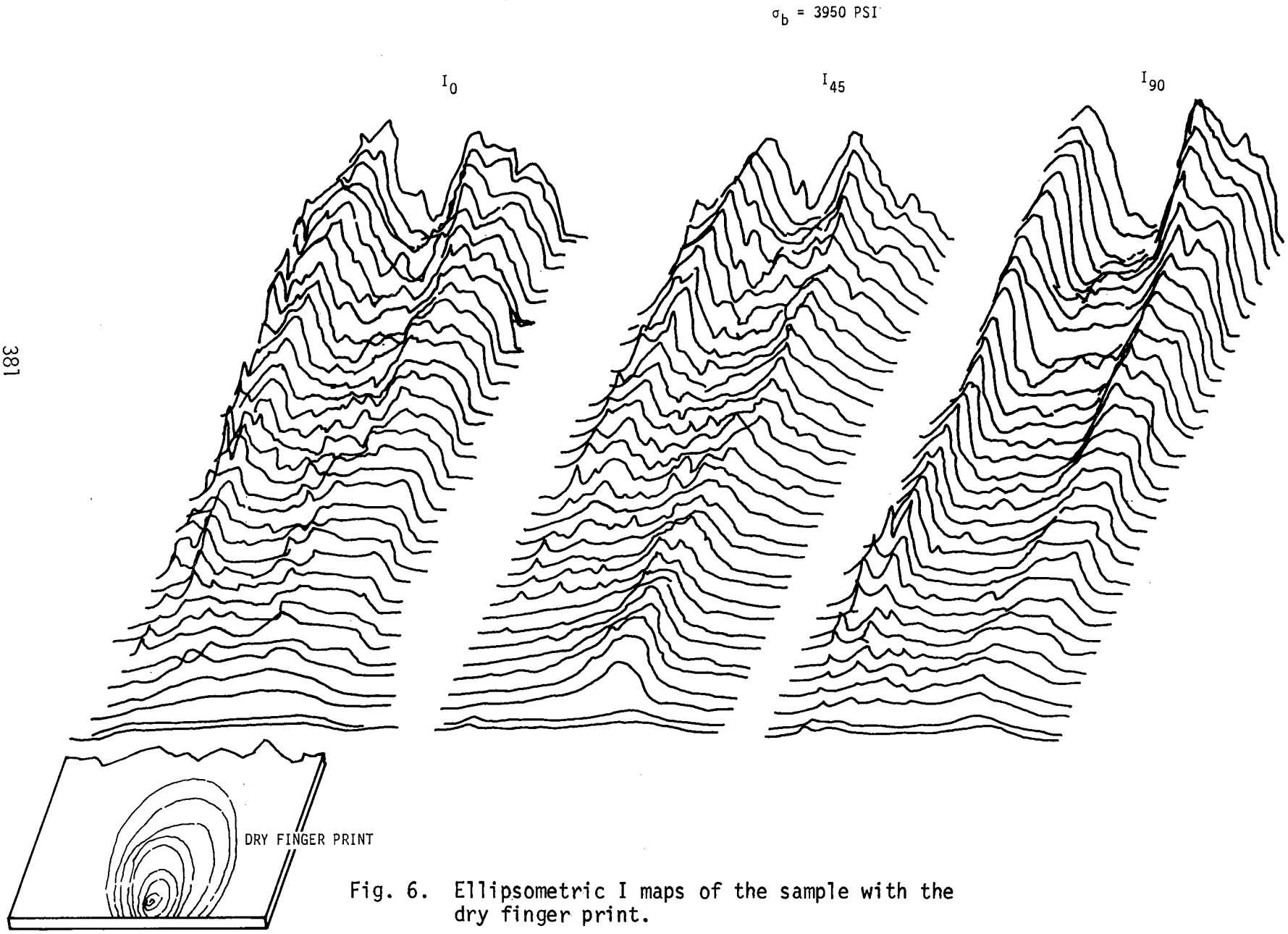



Fig. 6. Ellipsometric I maps of the sample with the dry finger print.

## DISCUSSION

DR. ANTHONY EVANS (National Bureau of Standards): Did the dry fingerprint and the fingerprint with the silicone grease show up any differently with any of those techniques?

DR. SMITH: Unfortunately, we haven't had a chance to map the greasy finger print yet. We just did the experiment to see if it would make a big difference in the bond strength at this point. The next step is to go ahead and redo that experiment. I am convinced that if we can see the dry fingerprint, we will be able to see the greasy fingerprint much more readily. I don't think there will be any problem but I can't show it to you because we haven't actually done it.

DR. WILLIAM SCOTT (Naval Air Development Center): These techniques of photo emission and ellipsometry, I realize, are very sensitive, and I almost question if they are not too sensitive. Very often in solid state literature you see someone performing an experiment at $10^{-10}$. torr that someone performed at $10^{-8}$ torr with completely different results. Considering the wide variety of things floating through the air, what are the chances that you will be able to get enough uniformity in materials that you could ever use it as a practical test?

DR. SMITH: Well, it is true, for example, in the measurements I just got through showing you that we actually depicted the fingerprint and it didn't make any difference to the bond strength, so you say we are overdetecting. More work will have to be done to show that in the actual problems in the factory that the kind of things that happen there will be shown up and that you won't have to be worrying about things that aren't going to be any trouble to you. I don't think it is going to be all that bad. I think it is going to work pretty well. Normally, you know, if you just leave your sample around the laboratory in the air, it gets some contamination from grease. You might get a bad bond from that. Less than 40 angstroms came from dipping the sample in water,

but it picked up enough to cause a bad bond, and that was depicted very well by this technique. I think that you do have a problem that you may be oversensitive in certain areas, but it is just a matter of getting enough research done to know where you are and what you are doing. That's the only answer I can give you. It is a lot better to be under that circumstance than the other way around where you can't detect anything.

MR. LEE GULLEY (Air Force Materials Laboratory): What is the current Rockwell method of control of the bonding surface prior to bonding? Can you give us some insight as to what is now?

DR. SMITH: That is too general a question for me to answer here because there are probably a dozen kinds of bonding that are going on at different parts of the company, and they are using all different procedures, and just what each one of them does, I couldn't answer.

MR. JOHN F. MOORE (B-1 Division, Rockwell International): I don't have any specific answers. I know as a result of degradation of bonds, there are currently specifications and controls and that there are variations in the adhesive strengths in the order of 25 to 50 percent. So we are quite interested in knowing what these variations are, but their effects are on the property and we are looking for an NDT method.